

according to definition (6), if one refers only to the menthol retained in the filter, or more exactly, the filter balance of the menthol entering the filter. As the values in Table 10 clearly show, the "true" menthol retention is considerably reduced with increasing pre-loading of the filter and in the 4th experiment, reaches the value of 0%. With still higher loadings, one obtains negative values for the "true" retention which are equivalent to a preponderance of elution.

The experimental cigarettes 1 to 6 contained different amounts of menthol in the rope. In order to eliminate this, we have related in Table 10 the amounts of menthol entering and leaving the filter, to the amount of menthol in the filter. With decreasing amounts of menthol in the rope, the percentage entering the filter decreases from 36 to 22%, presumably because of the increasing transfer of menthol to the subsidiary smoke stream. On the other hand, the percentage leaving the filter, which has also been given the name of smoke transfer, increases from 14% to about twice this value, since the filter, as has already been mentioned, becomes less effective with increasing loading. For the same cigarettes, the corresponding transfer values for nicotine are 17% (entry into filter) or 9% (exit from filter).

In a supplementary smoking experiment we were able to determine that there is a certain menthol elution also from the 8mm long tobacco stub immediately in front of the filter. For a menthol content of 1.71 mg/cig. in a rope of length 67 mm, the stub contained 0.20 mg/cig. before smoking and 0.16 mg/cig. after smoking.

With increasing storage time, the amount of menthol found in the smoke ^{even} increases in spite of the loss of menthol during the storage and despite the decrease in the percentage of menthol entering the filter because these effects are overcompensated for by the great decrease in filter efficiency resulting from

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the increasing pre-loading of the filter with menthol. As the length of storage increases, there is eventually a decrease in the amount of menthol in the smoke.

3.4 Comparative Analysis of Menthol Cigarettes

11 cigarette brands from the BRD and the USA, with different menthol contents, were analyzed for their menthol content, before smoking, in the rope and in the filter. They contained between 0.013 and 3.2 mg/cig. menthol in the whole cigarette. Apart from the two brands with very low menthol content, a menthol analysis was also carried out on the smoke and on the smoked filter. The average values two analysis each of the smoke and the tobacco are compiled in Table 11. The percentage of menthol in the filter varied between 23 and 42%, in most cases however between 30 and 37%. The age of the cigarettes was not known. In most cases it was however of the order of several months. ^{on}the effect of very different amounts of total menthol per cigarette on the percentage of menthol in the filter, is not discernible.

The amounts of menthol found in the smoke run parallel with the amounts of menthol found in the whole cigarette before smoking. This expresses itself, ~~for all brands~~, in the smoke transfer which is of the same order of magnitude for all brands, between 24 and 31%. The cigarette brand G is the only exception to this, having a smoke transfer of about 19%. Accordingly, Brand G had at 0.23 mg/cig., the lowest menthol content in the smoke of the actual menthol brands. This brand is different from all others because its cigarette paper is perforated over the whole length of the rope. With this cigarette, it is to be expected that a larger percentage of menthol than usual would go over to the subsidiary smoke stream. The Brand J on the other hand, which contains less than 10% of the

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Table 11. Comparative Analyses of Cigarette Brands

Tabelle 11. Vergleichende Analyse von Cigarettenmarken.

| BRAND Marke | BEFORE SMOKING | | | | AFTER SMOKING | | | | APPARENT | |
|----------------|---------------------------|--|----------------------|---------------------------------------|---------------------------|-----------------------------|---|-------------------------------------|--|-------------------------------|
| | ROPE Strang mg/Cig. | vor dem Rauchen FILTER Filter mg/Cig. | Cigarette mg/Cig. | Verfahren Anteil im Filter % | SMOKE Rauch mg/Cig. | FILTER Filter mg/Cig. | nach dem Rauchen SUM Summe mg/Cig. | schein- bare Re- tention % | FILTER Filter- bilanz mg/Cig. | Rauch- Über- gang %* |
| A | 2,11 | 1,06 | 3,19 | 33,9 | 0,63 | 1,12 | 1,75 | 84,0 | 0,04 | 29,9 |
| B | 1,85 | 0,86 | 2,81 | 30,8 | 0,58 | 0,88 | 1,44 | 81,1 | 0,02 | 28,7 |
| C** | 1,58 | 0,77 | 2,35 | 32,8 | 0,49 | 0,78 | 1,25 | 60,8 | - 0,01 | 31,0 |
| D | 1,70 | 0,57 | 2,27 | 25,0 | 0,42 | 0,64 | 1,06 | 60,4 | 0,07 | 24,7 |
| E | 1,29 | 0,77 | 2,05 | 37,3 | 0,36 | 0,76 | 1,14 | 66,7 | - 0,01 | 29,5 |
| F | 1,13 | 0,83 | 1,95 | 42,3 | 0,31 | 0,81 | 1,12 | 72,4 | - 0,02 | 27,5 |
| G** | 1,23 | 0,62 | 1,85 | 33,8 | 0,23 | 0,58 | 0,81 | 71,8 | - 0,04 | 18,7 |
| H | 1,00 | 0,56 | 1,56 | 35,9 | 0,31 | 0,52 | 0,83 | 82,7 | - 0,04 | 31,0 |
| J | 0,079 | 0,048 | 0,125 | 36,8 | 0,019 | 0,035 | 0,054 | 84,8 | - 0,011 | 24,1 |
| K | 0,013 | 0,008 | 0,019 | 31,6 | | | | | | |
| L | 0,010 | 0,003 | 0,013 | 22,8 | | | | | | |

Menthol in rope = 100%

***with perforations

**100 mm cigarettes(all others are 85 mm)

normal amount of menthol is, with 24% smoke transfer, in the normal region. The ^{amount of} menthol found in the smoke of this brand of cigarette, 0.02 mg/cig., is barely noticeable to an observant smoker.

For all the brands investigated, the "apparent" retention is between 60 and 72%, and thereby in the same range as our experimental cigarettes no. 1 to 6. A correlation with filter length (18 to 25 mm), the filter weight (120 to 165 mg), the filter resistance to drawing (53 to 75 mm column of water pressure) and the nicotine retention (35 to 48%) could not be detected, presumably as a result of the different ages of the cigarettes. The filter balance is between 0.07 and -0.04, i.e. the "true" retention and elution are in all cases nearly equally large.

Brands B and H contained menthone in addition to menthol. The amount was between 2 ^{or} and 4% of the amount of menthol given in Table 11, i.e. 6 or 12% (relative to the menthol) of pepper-

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mint oil were used in addition. Peppermint oil contains between 25 and 40% menthone.

SUMMARY

A method for the determination of menthol in tobacco, in filters before and after smoking as well as in the smoke of mentholated cigarettes is presented which is based on the combination of steam distillation and liquid-liquid partition followed by gas chromatography on polypropylene glycol in the presence of benzylpropionate as an internal standard. The procedure also permits menthone to be quantitatively determined as a characteristic constituent of peppermint oils. Analytical results concerning reproducibility, recovery and sensitivity of the method are detailed.

The transfer of menthol from the tobacco rod to the acetate filter and vice versa as a function of the storing period of mentholated cigarettes has been investigated as well as the menthol distribution due to the smoking process. A considerable selective retention of menthol by cellulose acetate filters could be demonstrated. Various mentholated cigarettes of different origin and of different menthol contents have comparatively been analysed.

LITERATUR

1. Carpenter, R. D., und Seligman, R. B.: 10th Tobacco Chemists' Res. Conf., Washington, 1956.
2. Lubus, J. J., und Bogue, H. D.: Tobacco Science 4 (1960) 213.
3. Massmune, H.: J. Biochem. Jap. 18 (1953) 277.
4. Mitchell, B. C., Barbee, E. F., und Irby, Jr., R. M.: Tobacco Science 7 (1963) 64.
5. Lyster, L. A.: Tobacco Science 11 (1967) 49.
6. DIN 10 240 (1969).

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